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A COMPARATIVE MULTI-TECHNIQUE INVESTIGATION ON MATERIAL IDENTIFICATION OF GILDING LAYERS AND THE CONSERVATION STATE OF 7 PORTUGUESE MANNERIST ALTARPIECES

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Abstract

This paper deals with the multi-analytical comparative characterization of 59 samples of gilded and polychrome layers from 7 altarpieces studied during the Gilt-Teller project (www.gilt-teller.pt). The altarpieces studied here belong to seven churches in the areas of Lisbon, Santarém, Portalegre and Guarda and display stylistic and constructive features characteristic to the Mannerism carved wood decoration in Portugal. The applied protocol of investigation characterized the structure and manufacture technique of gilding; identified the chemical composition of the layers constituting the gilded polychrome decoration; compared the materials and gilding techniques encountered in the 7 altarpieces and assessed the conservation state of each altarpiece. The analytical techniques applied to these purposes were: stereomicroscopy (SM), optical microscopy (OM), scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM-EDX), X-ray diffraction (XRD), MALDI-TOF mass spectrometry, µRaman and µFTIR/imaging µFTIR spectroscopies. This interdisciplinary multi-scale approach was used to elucidate the aspects related to the material and technical aspects of "talha dourada" decoration, answering to these questions: which are the original materials and layers in the making of the polychromy and which are the ones added with posterior interventions; which are the relationships between gilding materials and techniques, regarding the degree of erudition of each case study; which were the main causes of degradation and influence to their conservation condition?

Keywords: Altarpiece; Mannerist style; Portugal; carved gilded wood; gilding techniques and materials; comparative study; conservation state

Introduction

The definition of altarpiece (retable) comes from Latin: *retro* - behind, and *tabula* - table (panel), indicating the sacred place where the Eucharistic mass is celebrated and the panel behind the altar or around it, creating visual and spiritual motivations for celebrating the

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religious cult [1]. It is the place as excellence where the eyes of the faith are centered. For this reason the covering with gold leaf of retabular surfaces, sculptures or other artistic works served as symbol of magnificence and devotion shown by the artists with the purpose of worshiping God [2-3]. This form of art will create true schools where art craft masters specialized themselves in carving, gilding and applying the polychrome layers on the wood [3-4].

Mannerism affirms the preference for altarpieces with several floors, conceived under direct influence of the treaties of Palladio, Serlio or Vignola [1]. This type of retable can soon be found all over the Iberian Peninsula [5]. Typically, it presents long shapes, flat surfaces, as well as carefully minded effects that suggest inbalance and disproportion – in sum, the ideals of international Mannerism. In terms of ornamental composition, the elements typical of the mannerist style, are: garlands, geometric motifs and fruits, as well as angel figures.

For the Portuguese mannerism in sculptures and altarpieces in this period we should take into account the influence from the Spanish-Flemish inheritance rooted from the Spanish models of the sculpture schools from Valladolid, Sevilia or Madrid. The political and social conditions of the époque decisively have contributed to this, considering that Portugal was under the Castela's domination and the king was ruling from Madrid. Today it is known that there were various carvers/sculptors to work in Spanish churches, coming from cities such as Lisbon, Elvas, Portalegre and Campo Maior villas. The exchange of work force between the two countries is evident in this period, the migration of Portuguese artists to Spain being more frequent [6-7].

The present study takes into account six main altarpieces and one lateral from seven churches in the areas of Lisbon, Santarém, Portalegre and Guarda (Table 1, Fig. 1) [8]. Their structure of carved wood present elements specific to the Mannerist style of altarpiece: organization of the altarpiece in floors including paintings and sculptures, columns with grooved fust, presenting sometimes decoration in the third upper segment and *remate* in semicircular shape and sometimes a central medallion. In the case of the altarpiece from Linhares church some National baroque elements are also present. A short description of the altarpieces taken into study can also be found on the website of the GILT-Teller project [8] including also references on artists and characteristics of the style.

Presence or not Authorship of Church or of restoration Type of Period Region Acronym the carving/ chapel altarpiece intervention gilding work (Y/N) Portalegre Church of the Convent 16 - 17th Devotional PT-AM-SFCV of St. Francis century 1590 - 1592 Cathedral of Gaspar Coelho Narrative Portalegre (escultor e (1930; 1960) PT-AM-SPt entalhador) Guarda Church of the 17th Eucharistical and Immaculate century devotional (tabernacle door) Conception -parish PT-AM-NSCBç church of Baraçal Church of Our Lady of 17 - 18th Eucharistical and Assumption - parish century devotional (it has (1971)church of Linhares da National Baroque PT-AM-NSALs Beira elements) PT-AM-Church of the Convent 18th Y Eucharistical and CSAPnh (partial) of Anthony, Pinhel devotional century Santarém Church of Our Lady of 16 - 17th Narrative Ν Conception- parish century PT-AL-Ta church of Tancos 17^{th} Lisbon Church of St. Eucharistical Y PT-AM-SDBLx Domingos de Benfica (1980-1981)century

Table 1. General data on the sampled altarpieces



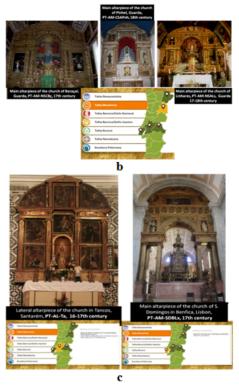


Fig. 1. General views of the seven altarpieces taken into study: a) from Portalegre district; b) from Guarda district; c) from Santarém and Lisbon areas

The study of these altarpieces aimed to answer to the following questions: which are the original materials and layers in the making of the polychromy and which are the ones added with posterior interventions or restorations; which are the relationships between gilding materials and techniques, regarding the degree of erudition of each case study; which were the main causes of degradation and influence to their conservation condition?

The different microscopic (OM, SEM), diffractometric (XRD) and spectroscopic (uRaman, $\mu FTIR$ and imaging $\mu FTIR$) and mass spectrometric (MALDI-TOF) techniques proposed for this study create a novel complementary approach aimed to characterize and compare the gilding materials and techniques [9-10], describing main features of the analysed samples from macroscopic to micro and molecular levels and the conservation state of the gilded and polychrome composites [11-12].

Experimental

Sampling

To study the materiality of the 7 altarpieces, 59 samples were taken (Table 2). The samples were taken from areas at the bottom front side of the altarpiece (known as *predela*), columns and from gilded carved wood decoration and polychromy of incarnations. The sampling criteria took also into account the lack of previous interventions of restoration (where possible) and possible presence of historical document attesting the data and authorship of the carving and gilding work.

The main data on the sampled altarpieces and the analyses performed on each of them are given in the Table 2.

Acronym	Nº of samples	OM-Vis, UV	SEM-EDS	XRD	μ Raman	μ FTIR/ imaging μ FTIR	MALDI- TOF- MS
PT-AM-SFCV	8	7	4	2	2	2	1
PT-AM-SPt	4	4	4	3	2	3	3
PT-AM-NSCBç	9	9	3	3	3	2	2
PT-AM-NSALs	6	6	3	2	1	2	2
PT-AM-CSAPnh	10	9	5	2	3	2	3
PT-AL-Ta	10	8	4	3	2	2	2
PT-AM-SDBLx	12	12	3	3	3	3	2

Table 2. Samples by altarpiece and performed analyses

Analytical Protocol

The analytical interdisciplinary approach, including microscopy (OM and SEM-EDX), X-ray diffraction, spectroscopy (microFTIR; microRaman) and MALDI-TOF spectrometry was applied for better understanding the structure, composition and conservation state of the gilded polychrome composites. The identification of organic constituents in these samples (binders, varnishes or other organic materials) was done by complementing the cross-section observation under optical microscope with microFTIR and MALDI-TOF MS identification.

Optical microscopy (OM)

The cross-sections, obtained from micro-fragments of the gilded samples, were embedded in Polyester resin Mecaprex SS (Leica) and properly polished after the necessary curing time. Their observation was done using an Axioplan Zeiss 2 imaging binocular microscope (50x–500x magnification), with both Visible and fluorescent light, and a Nikon DXM1200F digital camera. The filter blocks used for observing the fluorescence were f8 (G 365, FT 395 and LP 420) and f6 (BP 450-490, FT 510 and LP 515). Visible light observations (illumination position for dark field observation, abbreviated as f2) were performed in reflection geometry.

X-ray diffraction (XRD)

A part of the samples were analyzed using X-ray difractometer model Panalytical X'PERT PRO, with $CuK\alpha$ source. The measurement parameters were: 2θ (5–70°); step size 0.002° 20; counting time 5–15 s; generator operating at 35 mA and 40 kV. The identification of crystalline phases was done using the software High Score Plus and the database PDF2.

Other samples were analyzed using commercial Bruker D8 Discover System with the DAVINCI design with Cu K α source operating at 40 kV and 40 mA and LINXEYETM 1-dimensional detector was used. The samples were placed onto a flat zero-background sample holder and irradiated through 0.6 mm slit. The micro beam was achieved using Göbel mirror and 1 mm collimator. The angular range (20) was scanned from 3° to 70° at step size of 0.02° with counting time of 1 s/step. Evaluation of X-ray diffractograms was made by using the routines of the Diffrac. EVAsoftware package (Bruker/AXS GmbH, Germany) and the PDF-2 database files (ICDD, Denver, USA).

Scanning Electron Microscopy – Energy Dispersive X-ray Spectrometry (SEM-EDS)

SEM – VEGA II LSH Scanning Electron Microscope (TESCAN - Czech Republic), coupled with EDX – QUANTAX QX2 (ROENTEC – Germany) spectrometer was used. Quantax QX2 uses detector of third generation Xflash, that does not need cooling with nitrogen and is 10 times faster than traditional detector based on Si(Li). The EDS spectra have been acquired in the following conditions: 20 kV voltage; 1x10⁻³ Pa; 5000 nA; working distance 11–20 mm (16.6 mm for EDX), scanning speed: 200 ns; magnification: 78x and 1000x. The cross-sections were covered with a fine layer of graphite using a specific "sputter coater".

HITACHI S3700N interfaced with a QUANTAX EDS microanalysis system was also used to obtain the backscattering images, point analysis and chemical mapping data. The

QUANTAX system was equipped with Bruker AXS 5010XFlash® Silicon Drift Detector (129 eV Spectral Resolution at FWHM/Mn K α). Standardless PB/ZAF quantitative elemental analysis was performed using the Bruker ESPRIT software. The operating conditions for EDS analysis were as follows: backscattered electron mode (BSEM), 20 kV accelerating voltage, 10 mm working distance. The cross-sections were previously coated with graphite.

MicroRaman spectroscopy (µRaman)

For microRaman analysis a microspectrometer HORIBA XPlora equipped with 785 nm laser was used. The spectra were acquired in extended scanning mode, in the region 100–2000 cm⁻¹. The laser beam was focused with lens of 50x from Olympus, with laser power on the sample's surface of 1.1 mW (5 seconds of exposure, 15 cycles of accumulation).

Fourier Transformed Infrared Micro-spectroscopy (µFTIR) and imaging µFTIR

Three samples (PT-AM-SPt) were analysed using Nicolet Nexus spectrophotometer interfaced with Continuum microscope with liquid nitrogen cooled MCT-A detector. The spatial resolution was 30 µm, the spectra were obtained in transmission mode using a Thermo diamond anvil compression cell with resolution of 4 cm⁻¹ and 128 scans in an interval between 4000 and 650 cm⁻¹. For the rest of the samples, another instrument Bruker, model Tensor 27, was used in medium infrared region (MIR) coupled with a Hyperion 3000 microscope controlled by the software OPUS 7.2, Copyright© Bruker Optik GmbH 2012, with FPA (Focal Plane Array) detector, that allows the acquisition of spectral images in which each pixel corresponds to spectrum of small region on the sample. The cross-sections were analyzed in ATR (Attenuated Total Reflectance) mode using an ATR objective of 20x with a Ge crystal and a pressure sensor in position 1. The IR spectra were traced in the region 3900–900 cm⁻¹ with 64 scans and spectral resolution of 8 cm⁻¹.

Matrix-assisted laser desorption/ionisation-time of flight mass spectrometry (MALDI-TOF MS) Protein digestion and purification

 $20~\mu L$ of $50~mM~NH_4HCO_3$ containing approximately $10~\mu g/mL$ of trypsin was applied to the samples and let react at room temperature for two hours. After the trypsin digestion, the solutions were taken from the surfaces and purified on reverse phase ZipTip. After equilibrating, binding and washing steps, target compounds were desorbed from the stationary phase. The solutions were consequently used for analyses by MALDI-TOF MS.

An aliquot of the elution solution containing peptides (2 μ L) was mixed with 4 μ L of 2,5-dihydroxybenzoic acid in water (1/2 [v/v]). The part of the resulting mixture (2.8 μ L) was for two times spotted on the stainless steel MALDI target and dried in air.

Mass spectra were acquired by a Bruker-Daltonics Biflex IV MALDI-TOF mass spectrometer equipped with a standard nitrogen laser (337 nm) in positive reflector mode with a mass accuracy of 0.2 Da; at least 200 laser shots were collected for each spectrum. The spectra were analysed using XMASS (Bruker), mMass software and a homemade database of reference proteinaceous binders [13].

Results and Discussion

Characterization of gilding - polychromy stratigraphy

Generally the samples display a stratigraphic structure of layers typical for water-gilded composites [14, 16], including layers of gesso (gesso grosso and gesso fine), bole and leaf and in case of polychromy also one or two layers of color over the metal leaf or without it, directly over the gesso. The gesso has a coarse granulometry for the layer of gesso grosso and a finer one for gesso fine and in some cases a high porosity, more easily observed under SEM bse than under Vis-OM (Fig. 2).

The bole layers have usually a single color for the same sample/area but in few cases two different and overlapped colors were observed, such it is the case of samples from the lateral altarpiece in Tancos (PT-Al-Ta 3, 6 and 7), Figure 3. The lighter bole (yellow ochre) under the

reddish-orange layer one seems to have a less concentrated amount of aluminium-silicates and almost no Fe which could suggest that this bole is mainly based on kaolinite while the upper layers are more Fe-based.

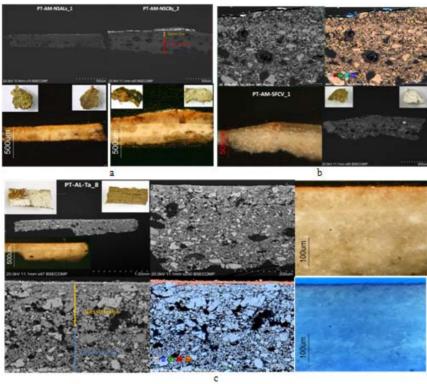


Fig. 2. Imaging of gesso layers using optical microscopy (OM) and scanning electron microscopy with backscattered electrons (SEM bse) for 3 samples: a) PT-AM-NSALs_1 and PT-AM-NSCBc_2; b) PT-AM-SFCV_1; c) PT-AL-Ta_8

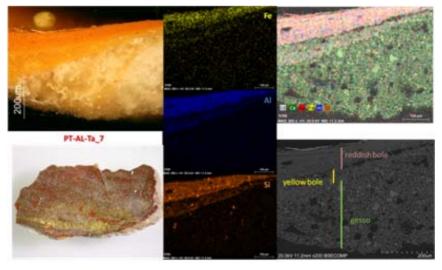


Fig. 3. Bole layers in sample 7 from the lateral altarpiece in Tancos (Santarém)

Estofado ou *esgrafito* decorations [15-16] were identified for altarpieces such as S. Domingo in Benfica (Lisbon) and main altarpiece of Pinhel (Guarda). Figure 4 shows the stratigraphic pattern of an *esgrafito* area, where the polychrome layer overlap the leaf applied over an yellow bole and white gesso ground.

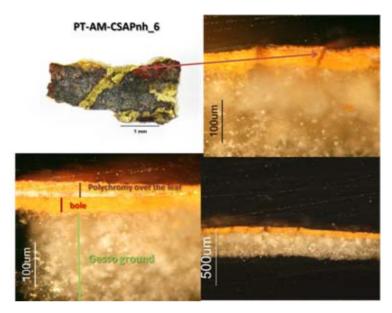


Fig. 4. Stereomicroscopy and optical microscopy observation of a sample from *esgrafito* area in the case of the main altarpiece from Pinhel, Guarda

Organic coatings were identified in only few cases, the UV-OM images showing an orange or bluish varnish for thin layers on the top of the gilding or in between layers in case of regilding areas or with reddish color (probably a lake mixed with some resinous varnish), figure 5.

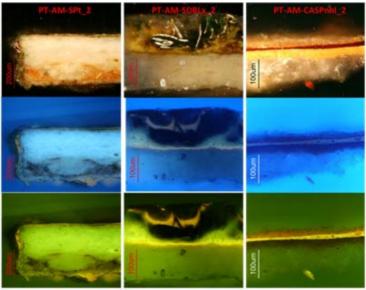


Fig. 5. Visibe and fluorescence imaging under optical microscope for 3 samples of 3 different altarpieces showing the presence of organic coatings displaying specific fluorescence pattern

Characterization of gilding materials and techniques

The ground layers are mainly made of gypsum and anhydrite (*gesso* - Ca, S, O) but in some cases *calcite* (Ca, C, O) in mixture with gesso was found in the samples from the following altarpieces: PT-AM-SDBLx (samples 2, 4 and 7); PT-AM-NSCBç (sample 2); PT-AM-SFCV (sample 2, aragonite identified also). Quartz (Qz) and silicates (Si, Al, K) were also detected in the case of altarpieces: PT-AM-SPt (sample 4); PT-AM-SFCV (sample 6); PT-AM-CSAPnh (samples 3 and 5).

Figure 6 shows the chemical identification by imaging microFTIR of the gesso ground in a cross-section made from a sample from the lateral altarpiece in Tancos.

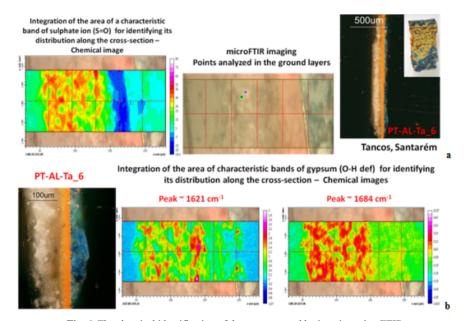


Fig. 6. The chemical identification of the gesso ground by imaging microFTIR in sample PT-AL-Ta_6

Bole layers are mainly composed of hematite (Fe oxides) and clay minerals (Al, Si, K), mainly kaolinite, but also illite and muscovite. In some cases quartz (Qz, for PT-AM-SPt, PT-AM-NSCBç,) and hydrocerussite in mixture with silicates and kaolinite (PT-AM-NSCBç_2) were found.

The leaf is generally represented by alloys of Au/Ag/Cu or Au/Cu/Ag varying the value of carat between 20-21 (altarpiece PT-AL-Ta) and 22-23 (all the other 6 altarpieces), Fig. 8a. In the case of the lateral altarpiece from Tancos a peculiar situation was detected, as the leaf applied over the columns is made of Au/Ag alloy of 23.86 karats while in the samples from other areas of *predela* the leaf has a lower purity (and more complex composition, including also Cu).

As substitute of the gold leaf we could also detect the presence of alloys of Cu/Zn (re-gilding areas) in the case of altarpieces: PT-AM-SDBLx (sample 10, Fig. 8b); PT-AM-SPt (sample 2); PT-AM-SFCV (sample 1); PT-AM-CSAPnh (sample 1) and Ag leaf for sample PT-AM-NSCBç 8.

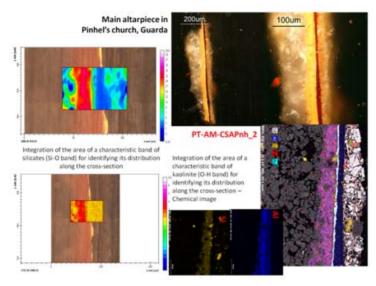


Fig. 7. Bole identification in a sample from main altarpiece in Pinhel using OM-Vis, microFTIR chemical imaging and SEM-EDS on cross-section

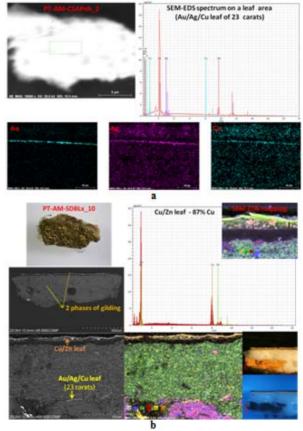


Fig. 8. Leaf identification using SEM-EDS for a) a sample from the main altarpiece in Pinhel (Guarda) and b) double gilding (Au leaf and Cu/Zn alloy) for a sample from the main altarpiece in S. Domingos em Benfica (Lisbon)

The main **binders** which were detected were: animal glue in the ground (Figure 9a) and bole layers and oil (Figure 9b) in some cases (Linhares, Baraçal, Tancos) for samples of polychromy. **The protective layers** identified in few samples only were wax as surface layer in some cases (Linhares) and shellac (Figure 9a) as varnish (PT-AM-SPt and PT-AM-SDBLx).

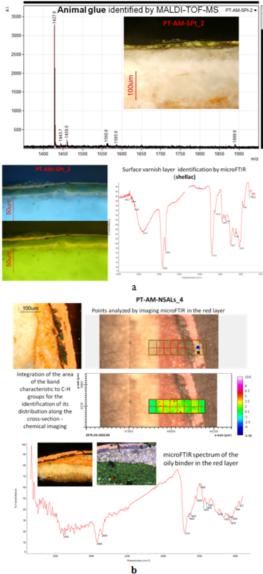


Fig. 9. Examples of identification of animal glue as binder in the ground layer and of shellac varnish as protective coating (sample PT-AM-SPt_2) and of oil for a red sample (PT-AM-NSALs_4) from the main altarpiece in Linhares (Guarda)

The polychromy is present in the flesh-tones of some characters (such as the Christ on Tabernacle's doors or sculptures within the altarpiece structures) or as decorative elements in red, green and blue tones. Figure 10 presents two cases of multi-technique identification of pigments and binders, one for flesh tones in a sample from main altarpiece in Linhares (Fig. 10a) and another one for blue layer over the gilding for 2 samples from the lateral altarpiece in

Tancos (Fig. 10b). Traditional pigments such as lead white (cerussite and/or hydrocerussite), anglesite, red ochre (hematite), minium or cinnabar, yellow ochres (goethite), azurite, sodalite were identified but also more recent ones (barite, chromium green) where posterior interventions of re-gilding or re-polychromy were detected.

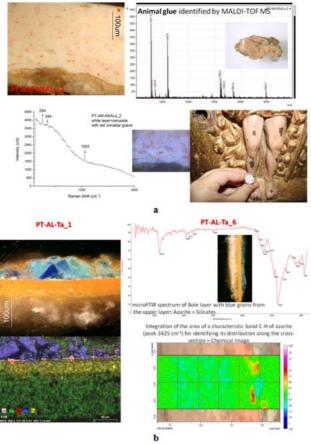


Fig. 10. Identification of cerussite and cinnabar in the flesh tone of sample PT-AM-NSALs_2 using microRaman (a) and of the azurite and silicates in the bole for samples 1 and 6 from Tancos using SEM-EDs and microFTIR imaging (b)

Conservation state assessment

Within the GILT-Teller project more than 35 churches on the whole territory of Portugal were visited and surveyed both for the gilded carved wood work and technical procedures [8-10], but also for their conservation condition [17]. A special technical sheet for the evaluation of the conservation state was designed and used for this survey [18]. Regarding the conservation state of the 7 altarpiece, in most cases the following degradation and deterioration pathologies were reported:

- Dust and soot depositions on gilded surfaces;
- Abrasion of wood surface and gilding;
- Fractures and disassembled elements:
- Craquelure;
- Disassembled and missing elements;
- Re-painting and re-gilding;
- Detachment of ground layers, gilding and polychromy;

- Lacunae of support and of gilding;
- Restoration interventions with replacement of missing elements with new materials;
- Punctual carbonization:
- Precarious state of the structure, visible behind the altarpiece;
- Biological infestation.

Figure 11 gives some examples of pathologies identified in 6 of the case studies (main altarpieces), while Figure 12 points out some of the pathologies using analytical techniques applied on samples.



Fig.11. Images with pathologies identified in the case of the 6 main altarpieces

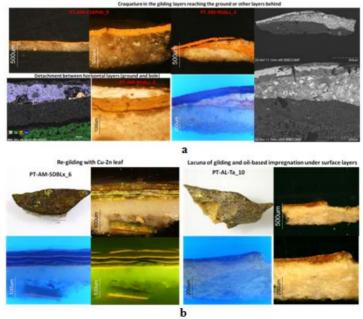


Fig.12. Cross-section observation under OM and SEM for identification of pathologies related with gilded structures/samples

Questions open to discussion

There are two aspects which need further clarification and study, such as:

1. Which are the relationships between gilding materials and techniques, regarding the degree of erudition of each case study?

The 7 altarpieces belong to different geographical areas in Portugal: *urban* areas (Lisbon and Portalegre) and also interior or *rural* (Santarém and Guarda). Considering the composition of gilding layers and techniques of application of the gilding materials it is very difficult to assess the differences of erudition mainly because the quality of materials and the craftsman's skills seem to have been on the same level. The authorship is known only in the case of the main altarpiece of the cathedral in Portalegre (Gaspar Coelho, carver and sculptor, 1590–1592) [6]. It is also known that the artists were very moveable, being called to several different places from North to the South of the country, to work mostly in partnership in a corporative way. That is the reason for not having known authorship of some of the altarpieces and very few treatises to explain the technology of gilding and polychromy.

2. Which were the main causes of degradation and influence to their conservation condition?

The environment without control is the main cause of degradation and deterioration of a work of art. The different factors of degradation (direct physical damage; theft and vandalism, fire, water, biological infestation and contamination, light, incorrect temperature and Relative Humidity, RH) can be usually controlled by Man. Neglecting is one of the worst factors of degradation and can be hardly avoided as should be part of a strategy for continuous conservation and education for preservation of cultural heritage. Among the 7 altarpieces the only one that at the date of sampling was not restored is the lateral altarpiece from Tancos (Santarem). All the other 6 suffered interventions of restoration (as in the case of main altarpiece from the Pinhel's church, Guarda) that stabilized some degradation processes but for further preservation the environmental parameters inside churches must be monitored to understand their fluctuations (mainly RH) and the way to prevent their influence on the altarpieces.

Conclusions

The proposed integrated multi-technique protocol to investigate the seven gilded altarpieces enabled to ascertain the similarities and differences in the structure, materials and techniques of the "talha dourada" samples.

Generally the stratigraphic structure of the samples is the classical one for *water gilding technique*:

- White-ochre layer of ground (around 250–500 microns) made of gesso (gypsum for gesso matte + anhydrite for gesso grosso) with inclusions of silicates and quartz; in few cases calcite was identified in mixture with the gesso;
- Bole of yellow or red /orange color made of Fe oxides, and clay minerals (e.g.

kaolinite, illite, muscovite); sometimes two different layers of different color (yellow and red) can be observed;

- Gold leaf (alloy of Au/Ag/Cu or just Au/Ag) of different purity (from 20 to 23 e even 23.86 carats) or Cu/Zn leaf and Ag imitations for few cases (see below);
- Polychromy of different color (white for flesh tones, blue or green for red for
 decorations alternating gold leaf and pigments or dyes) general use of lead white
 (cerussite/hydrocerussite) with sometimes calcite, anglesite and in few cases barite;
 cinnabar and red lead (minium) grains inside the white layers; goethite for yellow
 layers; azurite and sodalite for blue and green tones; chromium green in one case (San
 Domingos in Benfica altarpiece);
- Animal glue identified as main binder of ground and bole layers;
- *Oil* was identified in few cases (Tancos) in the silicate layers below the leaf;
- Varnishes present in some cases, *shellac* (associated with presence of re-gilding layers) being identified and also *wax* as protective layer.

Regarding the techniques of application, *gesso fine/gesso grosso* applications in the ground are present in most cases, only for few one type of gesso was used.

The posterior interventions identified were:

- Re-gilding in the case of altarpieces PT-AM-SDBLx (sample 10); PT-AM-SPt (sample 2); PT-AM-SFCV (sample 1); PT-AM-CSAPnh (sample 1);
- Re-polychromy (recent pigments, like barite and chromium green were found in the altarpieces of S. Domingos in Benfica, Baraçal, Pinhel, Tancos).

As a general view the altarpieces displayed the same characteristics of materials, composition of gilding and polychrome layers. The gold leaf had a slightly different percentage of Au in the alloy but with low impact on the final work. The conservation state can be appreciated as being medium to good in most of the altarpieces, only for the lateral altarpiece in Tancos the degree of decay can be defined as "bad condition". The RH inside the churches was very similar, cold and damp in winter time and drier but also cold in summer time. These conditions were a factor of deterioration of the ground layers, gilding and polychromy leading to cracks networks (craquleure), detachments in between layers and from support, lacunae and missing elements, fractures and fissures in the wooden support etc. These environment conditions were also favourable to the biological infestation, especially the wood boring insects attack.

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